



Dye extraction from *Rheum emodi* for colouring silk using natural mordants

M. Srivastava¹, D. Mogra^{1*} and P.Gupta²

¹Department of Textiles and Apparel Designing, College of Home Science, Maharana Pratap University of Agriculture & Technology, Udaipur –313001 (Rajasthan), INDIA

²Department of chemistry, Agra College, Agra - 282004 (UP), INDIA

*Corresponding author. E-mail: dollymogra@gmail.com

Received: November 14, 2014; Revised received: March 2, 2015; Accepted: March 30, 2015

Abstract: Applicability of natural dyes has increased several folds in the past few years due to the eco-friendly approach of the people. This paper concerns with the dyeing of silk fabric with colorant extracted from *Rheum emodi* (commonly known as rhubarb, himalayan rhubarb, Indian rhubarb) in the presence of selected five natural mordants. Extract using hot percolation with methanol was (27.55%) found suitable and maximum absorption (λ_{\max}) was observed at pH of 10 in aqueous solution. A wide range of shades ranging from yellow to olive green were obtained. As per the visual evaluation Concentration (0.1%), dyeing time (60 min.), mordant concentration (2%) and pre mordanting method were observed optimized for dyeing. From the results of fastness grade for sunlight and washing it is concluded that the samples mordanted with Harda (*Terminalia chebula*) and Amla (*Phyllanthus emblica*) were excellent followed by Anar (*Punica granatum*), Bahera (*Terminalia bellerica*) and Katth (*Acacia Catechu*).

Keywords: Extraction, Mordants, Natural, *Rheum emodi*, Silk

INTRODUCTION

Natural dyes have better biodegradability and generally have higher compatibility with the environment. They are nontoxic, non-allergic to skin, non-carcinogenic, easily available and renewable (Adeel *et al.*, 2009; Pruthi *et al.*, 2007; Onal, 1996). Natural dyes have been a part of human life since time immemorial and are obtained from plants, minerals and animals, According to the chemical structure, the natural plant dye can be divided into indigo type, anthraquinone type, flavone type, alkaloids type, multi-hydroxybenzene type, diketone type, benzopyran type, and carotenoid type (Guizen *et al.*, 2008). Natural dyes are non-toxic and do not create environmental problems due to their bio-degradable nature (Kulkarni *et al.* 2011). Demand of natural dyes is increasing continuously (Samanta and Agarwal, 2009) as their production and application does not require strong acids and alkalies (Bhuyan *et al.*, 2004). Rhubarb is the name given to different species of *Rheum* (Family Polygonaceae) growing wild in the temperate and subtropical region of the world chiefly in Asian countries, viz. India (Kashmir, Assam, Sikkim), Nepal, Bhutan, China, Pakistan, Turkey and Tibet. Rhubarb is a perennial plant, with large fleshy rhizomes, large leaves and long thick petioles (stalk). Several species of Rhubarb are used in medicine, some for culinary purpose and few are grown as ornamentals plants. There are three types of Rhubarb, viz. the Chinese Rhubarb, the Indian or Himalayan Rhubarb and the Rapontic Rhubarb (Khan *et al.*, 2004). *Rheum emodi*,

commonly known as dolu, is a stout herb of 1.5-3 m in height, distributed in the Himalayas from Kashmir to Sikkim at an altitude of 3300-5200 m. The stout roots are the chief source of Himalayan rhubarb and finds application chiefly in medicine as a purgative and astringent tonic and can also be used for colouration of wool and silk fibre (Maulik and Roy, 2009).

Indian Rhubarb almost odorless or somewhat fragrant, with bitter astringent taste. It contains anthraquinone derivatives and yield about 25 to 30 percent of extractive soluble in 50 percent aqueous alcohol (Agarwal *et al.* 2001). *Rheum emodi* is a storehouse of a large number of anthraquinone derivatives which are known for a large number of biological activities of significant potency (Sharma *et al.*, 2012). Colouring component of *R. emodi* has close resemblance with a typical anthraquinonoid type disperse dye; the uptake of such colouring component by the protein fibres remains unaffected within a wide pH range of 4-8, colour uptake, rate of dyeing and affinity of colour are found to be more for silk (Das *et al.*, 2008). Indian rhubarb contains a number of anthraquinone derivatives based on emodin, emodin-3-monomethyl ether, chrysophanol, aloe-emodin and rhein. These occur free and as quinine, anthrone or dianthrone glycoside (Shah & Seth, 2010). The alkaline extract gives yellow, brown and grey shades of good fastness on mordanted substrate (Sengupta, 2007).

It has been found that required scientific studies and systematic reports on dyeing of textiles with natural dyes are still insufficient (Koyuncu, 2012). In order to strengthen the use of natural dyeing with natural mordants

the present study is aimed at applying the colouring component of *Rheum emodi* on silk fabric for establishing optimum conditions of dyeing (like dye bath pH, dye concentration, dyeing time, mordant concentration, mordanting method) and assessing different colourfastness properties. Wavelength of maximum absorption and suitable percolation method was also enlisted for *R. emodi*.

MATERIALS AND METHODS

Selection and extraction of natural dye: Clean, dry rhizome of *R. emodi* were dried under shade and pulverized to powder form and used throughout present study. The powdered material (400g) was subjected to hot percolation in soxhlet apparatus and extracted with methanol (2L). The extraction was continued till the entire colour was extracted. The solvent was removed under reduced pressure to yield a thick syrupy extract which was used for dyeing of silk. The percentage extractive of methanol extract using hot percolation with methanol was 27.55% which was more than cold percolation 23.9%, so hot percolation was preferred for extraction of dye from powdered rhizome of *R. emodi*.

Procurement of fabric: A pure white Degummed, bleached mulberry silk fabric weighing 50gm/sq.m was procured from Gandhi Ashram shop in the local market of Agra.

Scouring of fabric: It was scoured with 2g/l non-ionic detergent at 50°C for 1 hour keeping the M: L ratio of 1:50 to remove fatty impurities, washed with water and dried under shade and ironed.

Selection of natural mordants: Myrobalan or Harda fruit skin (*Terminalia chebula*), Anar rind (*Punica granatum*), Amla (*Emblica officinalis*), Bahera (*Terminalia bellirica*) and Kattha (*Acacia catechu*) were used as natural mordants and procured from local market of Agra.

Mordanting methods: The three methods of mordanting namely pre-mordanting (followed by dyeing), post-mordanting (dyeing Followed by mordanting), and simultaneous mordanting (dyeing and mordanting together) were used.

Pre-mordanting: In pre-mordanting method, the fabrics were first immersed in an aqueous solution of mordants for 60 min at 70°C and rinsed thoroughly with water. The mordanted fabrics were then dyed at 70°-80°C for 1 hour.

Simultaneous mordanting: In simultaneous mordanting, the silk samples were immersed in a bath containing both mordant and dye solution together. The dyeing temperature was kept at 70°-80°C for 1 hour.

Post mordanting: In case of post- mordanting, the dyeing was carried out in the absence of mordants at 70°-80°C for 1 hour and rinsed with water. Dyed fabrics were then mordanted in a separate bath at 70°C for 60 min.

Neutralization of dyed samples: The dyed samples were neutralized by using various concentrations (0.5,

1 and 2%) of dilute acetic acid.

Colour fastness: The color fastness is usually rated either by loss of depth of color in original sample or is expressed by staining scale (Samanta and Agarwal, 2009). The dyed samples were subjected to sunlight and washing fastness test to find out the resistance of colours to light and washing. When the samples were exposed the staining properties of the dye were also studied.

Instruments used: To optimize the variables of dyeing and to assess the wash and light fastness the following equipments and other materials were used-

a) SL-171 colorimeter from Elico India Ltd. was used to find out λ_{\max} and optical density of the dyes.

b) Exposure rack, Blue wool standards and opaque cover were used for colour fastness to sunlight of dyed samples.

c) Launder-O-meter, detergent and Grey scale were used for colour fastness to washing of dyed samples.

RESULTS AND DISCUSSION

Wavelength of maximum absorption: Table 1, reveals wavelength of maximum absorption (λ_{\max}) and observed colour of aqueous solution of *Rheum emodi* in absence and presence of acid, alkali and mordanting agents. In the absence of any of the above agents, aqueous solution of this dye gives a pH value of ~ 6.0 with a yellowish brown colour, showing its λ_{\max} at 408 nm. In the pH range of 2 - 6 there is no shifting of λ_{\max} value. But with the increase in pH of the aqueous solution, a bathochromic shift in wavelength of maximum absorption (λ_{\max}) towards higher wavelength is observed, giving λ_{\max} value of 454 at pH of 10.

Osman (2014) also computed that printing with rhubarb nanoparticles the optical absorption spectra is in visible region. It can be seen that, the unground rhubarb particles show two main absorption bands around 444 and 411 nm that are characterized by a red brown colour. On the other hand, rhubarb particles reveal a strong change of their optical absorption when their size is reduced. The spectra of ground rhubarb particles show blue shift of the main bands (to lower wave length) and appear at 440 and 400 nm.

The colour of the aqueous solution also changes steadily from yellowish-brown to brown-red in consequent to such shifting of λ_{\max} with the increase in pH value of aqueous solution. Under alkaline condition there is also an increase in absorbance value; this may be due to the ionization of hydroxyl group of the dye in alkaline medium, which increases aqueous solubility of such dye.

Optimization of dyeing conditions

Dye material concentration: The methanol extract of *Rheum emodi* was used for dyeing of silk fabric. Dyeing solution was prepared by dissolving ten gm of methanol extract of *R. emodi* in one liter solution of sodium carbonate (1%). This 1% solution of dyeing

Table 1. Wavelength of maximum absorption of aqueous solution of *Rheum emodi*.

Aqueous solution of <i>R. emodi</i> treated with	pH	Wavelength of maximum absorption nm	Colour observed
None	6	408	Yellowish brown
Acetic acid	4	402	Yellowish brown
Hydrochloric acid	2	392	Yellow
Sodium carbonate	8	440	Brown red
Sodium carbonate	10	454	Dark brown

Table 2. Sunlight and washing fastness for natural mordants.

S. No.	Dye Percentage	Properties		
		Sun Light	Washing fastness	
			CC	CS
1.	Rheum dye 0.1 %(without mordant)	5	3/4	3/4
2.	Amla 2% + Dye 0.1%	7	3/4	3/4
3.	Anar 2% + Dye 0.1%	5	4/5	3/4
4.	Bahera 2% + Dye 0.1%	4	4	3
5.	Harda 2% + Dye 0.1%	7	4/5	3/4
6.	Kattha 1% + Dye 0.1%	4	4	4

material was further diluted to prepare different concentrations of dye material. The optimum concentration of dye material was found out by taking varying concentration of dye material (0.02%, 0.05%, 0.1%, 0.15%, 0.2%) in a beaker and by dyeing the silk samples at 70⁰-80⁰C for one hour in it. During the dyeing process, the samples were constantly stirred to obtain an even dye on samples. The silk samples were taken out from the beakers, dried in shade and evaluated visually by panel of judges. On the basis of luster, depth of shade and overall appearance the best color was obtained at 0.1% concentration.

Dyeing time: Optimum time for dyeing was found out by taking three silk samples into three beakers containing dye solutions. The dyeing was carried out for (30, 60, 90 minutes). The samples were taken out from the beakers dried in shade and evaluated visually by panel of judges, on the basis of luster, evenness of dye, depth of shade and overall appearance. The best shade of colour was obtained by dyeing the samples for 60 minutes.

Mordant concentration: Different concentrations (1, 2, 3 g/100ml) were prepared by dissolving appropriate amount of natural mordants in water. The solutions were filtered to remove the impurities and the clear solution was used for mordanting of silk samples at 70-80⁰ for one hour, dried under shade and used for dyeing. The mordanted samples were evaluated visually by panel of judges, on the basis of luster, evenness of dye, depth of shade and overall appearance. The best shades of colour were obtained by 2% for natural mordants (except Kattha, 1%).

Neutralization of dyed sample: Out of various concentrations (0.5, 1 and 2%) of dilute acetic acid, the best results were obtained by using 1% on the basis of visual inspection.

Mordanting method: The best shades of colour were

obtained by pre-mordanting method.

Shades obtained on silk fabric: An assortment of colours obtained by pre-mordanted silk samples with various mordants followed by dyeing with *R. emodi* dye is shown below.

Nayak (2014) stated about colour analysis in terms of lightness (ΔL^*) and the Cartesian coordinates (Δa^*) (Δb^*) and showed that the overall principal colours yielded on jute-cotton and jute-wool union fabrics were ranged from yellow ochre, yellowish brown, deep brown, grey, and coffee to olive black and affected by the type of mordant used. Nayak also concluded that the type of shades produced and the level of absorption of colorant were affected by using the different mordants and pretreatment.

Colour fastness: The colour fastness to light and washing for silk samples dyed with *Rheum* dye in absence and presence of different mordanting agents is shown in table 2.

From the table 2, it is evident that the sample mordanted with natural mordants such as Harda and Amla exhibited excellent (7), Anar exhibited good (5) and Bahera and Kattha exhibited fair to good (4) colour fastness to sunlight.

As regard to colour fastness of silk samples to washing, the sample mordanted with natural mordants such as Harda and Anar exhibited very well to excellent (4/5) colour fastness properties to washing, very good for Kattha and Bahera (4) and good for Amla (3/4).

From the above result of fastness grade it has been comprised that the Harda mordanted sample exhibited excellent fastness property to sunlight and washing both, in all cases the colour fastness levels are quite satisfactory for practical application purposes where as they range between fair good to excellent.

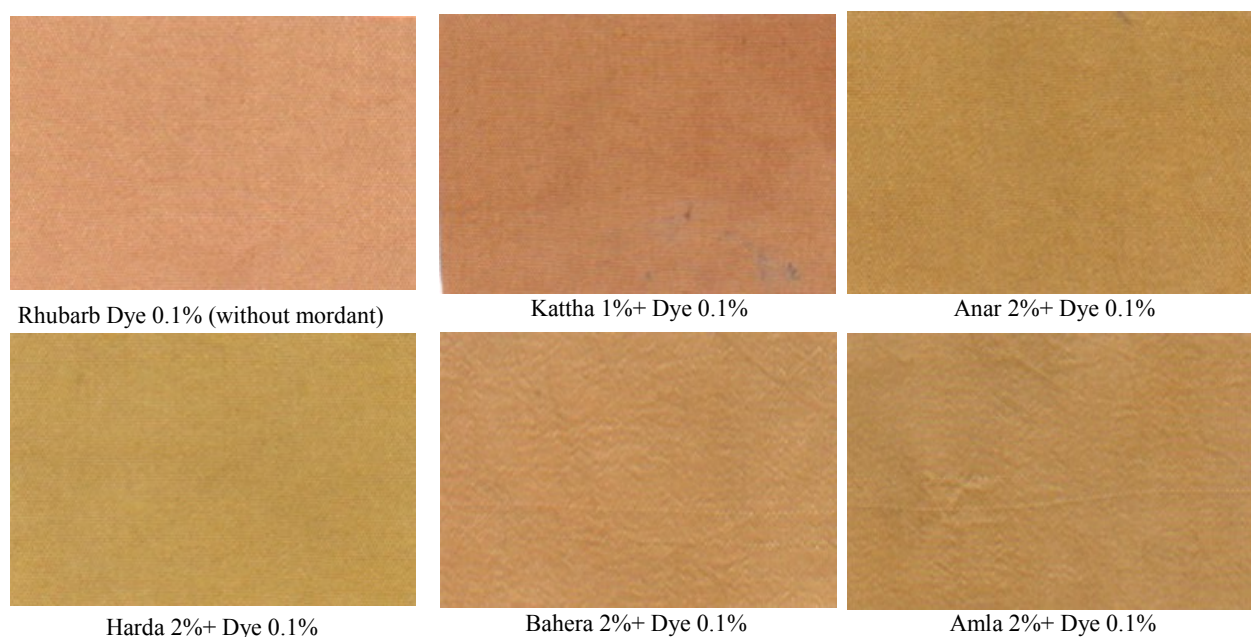


Fig. 1. Shades of dye and mordant combinations.

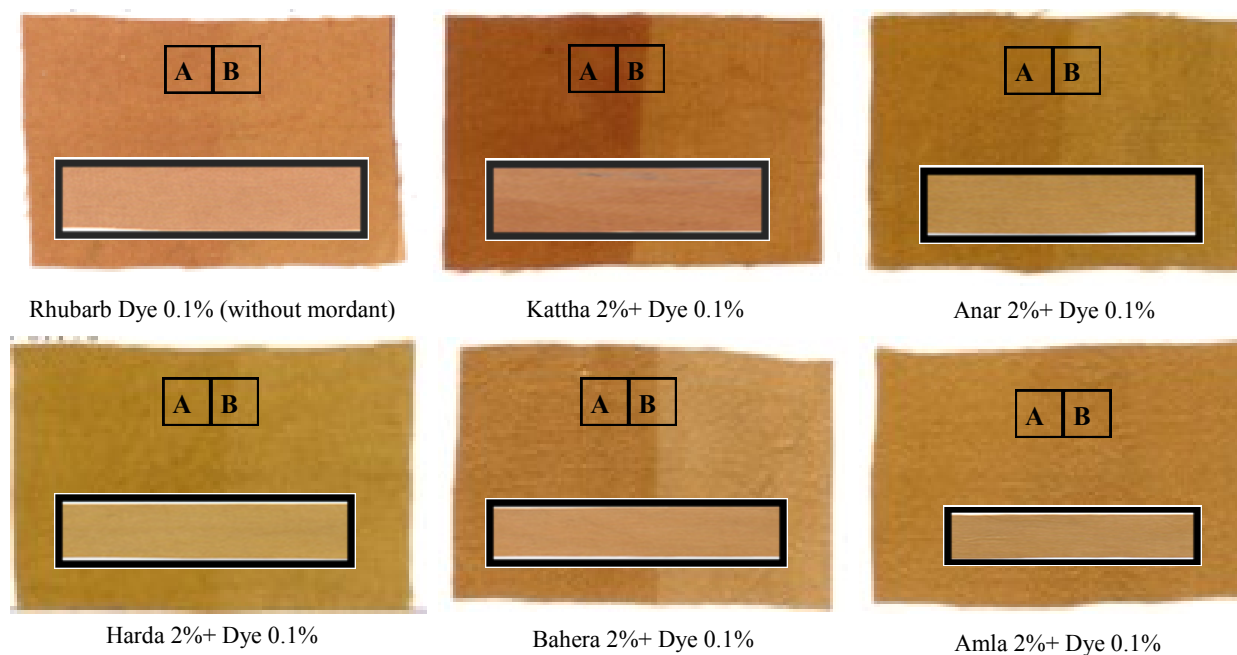


Fig. 2. Comparisons of pure samples (in box) with after treated samples of Sunlight (A) and Wash fastness (B).

Conclusion

The present study concluded that a range of colours were successfully obtained for dyeing of silk fabric using rhizomes of *R. emodi* with natural mordants. In view of significant promotion of natural dyes; dyeing conditions were optimized with respect to extraction, concentration, dyeing time, mordanting method along with wave length of maximum absorption. It was noteworthy that the washing and light fastness of natural dyed silk fabric in general was good for all the mordants. It is however recommended that the technique, other parameters and combinations will be further optimized

to enhance the applicability of *Rheum emodi* and other natural dye sources.

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